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HYDROGEN EMBRITTLEMENT OF A CYCLICALLY DEFORMED HIGH STRENGTH A--ETC(U)  
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RENSSELAER POLYTECHNIC INSTITUTE  
TROY, NEW YORK

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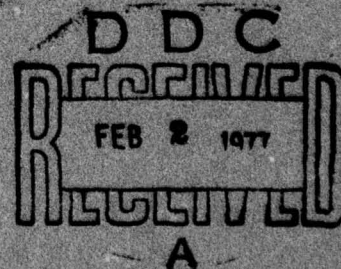
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HIGH STRENGTH Al ALLOY

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Abstract

High cycle fatigue experiments have been performed on a 7075 Al alloy, principally in the T6 temper in dry air, distilled water 0.5N NaCl and 0.5N Na<sub>2</sub>SO<sub>4</sub> solutions as functions of pH, cathodic charging and catalyst poisoning of the hydrogen evolution reaction. All aqueous solutions appreciably lowered fatigue resistance with Cl<sup>-</sup> ion producing the greatest reduction in resistance and SO<sub>4</sub><sup>=</sup> ion behaving essentially in the same manner as distilled water. Under cathodic charging conditions fatigue resistance is significantly reduced and both Cl<sup>-</sup> and SO<sub>4</sub><sup>=</sup> solutions produce similar fatigue lives. Acid solutions reduce fatigue resistance to a greater degree than do basic solutions of equivalent corrosivity and a catalyst poison (As) added to Cl<sup>-</sup> solutions reduces fatigue resistance relative to neutral Cl<sup>-</sup> solutions. Fractography of specimens fatigued in aqueous environments shows that a significant amount of cleavage and quasi-cleavage occurs, the extent of these features being apparently a function of available hydrogen to the alloy free surface and to the tips of growing cracks. On the basis of these observations,



it is suggested that corrosion fatigue of 7075 alloy is essentially a hydrogen embrittlement phenomenon where the low diffusivity of hydrogen is counterbalanced by the fact hydrogen need only be present in the alloy free surface for crack initiation and in the plastic zone of growing cracks for propagation.

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Introduction

Environmentally accelerated fatigue crack initiation and growth have been the subject of a considerable amount of recent research<sup>1-9</sup>. Mechanisms for this phenomenon, particularly in chloride containing solutions, have included active dissolution of active slip bands and surface energy reduction at crack tips due to preferentially adsorbing environmental species<sup>4-6</sup>. The possibility of a hydrogen embrittlement phenomenon was also suggested as one of several possibilities to explain reduced fatigue resistance of aluminum alloys exposed to water vapor<sup>7</sup>.

In previously reported work it was shown that corrosion fatigue crack initiation of 7075 Al alloys may be associated with hydrogen, generated by corrosion at non-metallic inclusions which intersect the alloy surface<sup>8</sup>. Similarly, initial crack growth was associated with hydrogen generated in an acid environment in a growing crack which acts as a crevice. Additionally it was shown that these effects are due to hydrogen contained in the alloy lattice, in contrast to surface energy reduction at a crack tip, since fatigue resistance of pre-charged specimens could be improved by a post-charging heat-treatment which effectively "baked" the



hydrogen out of the alloy . It was also shown that simultaneous cathodic charging of a cyclically stressed high purity analogue of the alloy (Al 5Zn 2.5Mg) resulted in very low fatigue resistance with crack surfaces showing a cleavage-type appearance. Greater reductions in fatigue resistance and large numbers of secondary cracks were observed for more negative cathodic potentials .

The results of the present study are part of a continuing effort to assess and understand the roles of hydrogen on the fatigue resistance of high-strength precipitation-hardened aluminum alloys.

#### Experimental Procedure

Axial fatigue experiments have been performed on cylindrical cross section (2.2mm) specimens of a commercial 7075 alloy in the T6 heat-treatment in dry air, 0.5N NaCl (as f(pH)), 0.5N Na<sub>2</sub>SO<sub>4</sub> and in 0.5N NaCl solution with addition of 10ppm As. For some experiments specimens were cathodically charged to produce hydrogen. Additionally some experiments were performed on specimens in the T73 condition (which has been reported to show superior SCC resistance) in dry air and in 0.5N NaCl solutions. Fatigue loading was in the tension-tension mode with a constant applied mean stress of 172 MPa ( $\approx \sigma_y/3$ ) under load control of 30 Hz. The environmental test apparatus has been previously described . Prior to testing, specimen surfaces were mechanically



polished to 3 $\mu$ m diamond paste and subsequently electropolished.

Solutions were aerated and recirculated from an external reservoir during testing. Fracture surfaces and the specimen free surfaces for each case were examined by optical microscopy and SEM.

### Results

Figure 1 shows the fatigue behavior of the alloy in air, distilled water, 0.5N Na<sub>2</sub>SO<sub>4</sub> and 0.5N NaCl and shows that, except at relatively low applied stresses, H<sub>2</sub>O and Na<sub>2</sub>SO<sub>4</sub> solutions show approximately the same fatigue behavior. Exposure to NaCl solution during cyclic stressing, on the other hand, results in very inferior fatigue resistance with the 10<sup>7</sup> cycle fatigue life corresponding to an applied cyclic stress of only about 35 MPa. A static loading test in NaCl, at an applied stress of about 320 MPa (a  $\sigma_{\text{mean}} + \sigma_{\text{cyclic max}}$  of this magnitude would result in  $N_f = 3 \times 10^4$  cycles) showed no evidence of cracking for a period greater than 200 hours.

Figure 2 shows the results of corrosion tests on 7075-T6 aluminum as a function of pH (controlled by NaOH or HCl additions) and indicates a minimum in corrosion rate in the pH 6-8 range. This data is similar to that reported for pure aluminum<sup>10</sup> in H<sub>2</sub>O + H<sub>2</sub>SO<sub>4</sub> or NaOH. Except that for the pure metal, the lowest corrosion rate has been reported to approach zero and, in general to be considerably lower for all pH's.

Figure 3 shows the S-N behavior for the alloy in a neutral solution and at pH 2 and 10.2 where the overall corrosion rates are approximately equal (about 100mdd). The data of Fig. 3 show that there is not a direct correlation of fatigue behavior with overall corrosion rate, pH 2 solutions reducing the  $10^7$  cycle fatigue limit to a value of about 15MPa. Additionally, there is a divergence of the data at higher applied stresses with the pH 10.2 solution showing longer lines than either the pH 6 solution or the pH 2 solution. At low applied cyclic stresses the pH 6 and pH 10.2 solutions produce approximately the same fatigue resistance while the pH 2 solution shows an almost constant percentage decrease in fatigue resistance when compared to the neutral solution. In an earlier paper, it was suggested that the reduction in fatigue resistance of high strength aluminum alloys was associated with hydrogen (which was produced by selective corrosion in the vicinity of non-metallic inclusions). The pH results also suggest a hydrogen interaction since pH 2 solutions result in lower fatigue resistance. In order to test this hypothesis, fatigue curves were generated for specimens which were maintained at a potential of about 1300 versus the saturated calomel electrode (about 600 mv cathodic to the corrosion potential). The cathodic current density at this potential corresponded to  $\approx 0.7 \text{ ma/cm}^2$  which could be expected to produce  $\approx 10^{15}$  molecules  $\text{H}_2/\text{cm}^2\text{sec}$ . (assuming that the measured



current is only used to generate  $H_2$ ). Figure 4 shows that, at high applied cyclic stresses cathodic charging results in an increase in fatigue resistance, while at lower applied stresses, cathodic charging results in decreased fatigue resistance with the  $10^7$  cycle fatigue limit reduced to a value of only  $\approx 13$  MPa. Charging at lower potentials does not appreciably affect fatigue resistance, although charging at higher potentials (less hydrogen evolved) results in some cathodic protection, a maximum in protection being observed at -900 mv vs. the saturated calomel electrode. Anodic polarization results in a still larger reduction in fatigue resistance (Fig. 5). The addition of a hydrogen atom recombination poison (10 ppm As) to the sodium chloride solution also results in a small reduction in fatigue resistance (Fig. 6).

Cathodic charging of the alloy exposed to neutral 0.5N  $Na_2SO_4$  results in a large reduction in fatigue resistance in a similar manner to cathodic charging in NaCl solution (Fig. 7). At stresses in the vicinity of the  $10^7$ -cycle fatigue limit, cathodic charging in sulfate results in virtually the same fatigue resistance as is observed in chlorides. At higher stresses, however sulfates are slightly less aggressive than chlorides.

In addition to experiments on the 7075 alloy in the T6 condition, a limited number of experiments were conducted on specimens in the T73 condition, a heat-treatment which has been shown to be more resistant to stress corrosion cracking. Figure 8 shows the results of these experiments with an expected slight



slight decrease in fatigue resistance in dry air (YS and UTS are lower for T73 than they are for the T6 heat treatment). In aerated 0.5N NaCl solution, however, there is no appreciable difference in fatigue resistance.

SEM fractography of specimens exposed to dry air, free corrosion in NaCl and under cathodic charging conditions are shown in Fig. 9. As has been previously reported<sup>8</sup>, in dry air the fracture surface consists of feathery river lines extending from a single initiation site at the alloy surface consistent with crystallographic (Stage I) cracking. In NaCl solution a featureless, thumbnail-shaped area which is normal to the tensile axis is associated with the initiation site. As the crack grows out from this area, straight cleavage-like markings develop which are unlike the feathery river lines observed in dry air in that they are straighter and more well defined. These markings are followed by the more traditional river line structure as the crack progresses, although the striations which are observed at longer crack lengths are of the "brittle" type observed by Forsyth<sup>2</sup> in contrast to "ductile" striations observed in air. In Na<sub>2</sub>SO<sub>4</sub> solutions, the featureless region and the cleavage like area is smaller than that observed for NaCl solutions (Fig. 10-a) "ductile" striations are observed near the overload failure (Fig. 10-b). The featureless and cleavage like zones observed after failure in solutions of pH 2 and pH 10.2 are

similar to those observed in neutral solutions (Fig. 11). Surface pitting, associated with inclusions, was also observed in the pH 2 solution although no secondary cracks were observed to be associated with pits (Fig. 12). Cathodically polarizing specimens to -1300mv vs SCE in NaCl solutions also resulted in the production of a featureless plus cleavage zone and, additionally a significant amount of Stage II, or non-crystallographic crack propagation was observed in regions far from the crack initiation site (Fig. 13-a). Polarization to -900mv vs. SCE also resulted in some grain boundary attack in regions removed from the initiation site (Fig. 13-b). Additionally, for cathodic potentials less than -900mv vs. SCE in either NaCl or  $\text{Na}_2\text{SO}_4$ , extensive secondary cracking and associated pitting was observed on alloy surfaces (Fig. 14-a). Optical metallography of cross-sections of the free surfaces indicated that the pits were associated with the cracks rather than being responsible for their formation due to stress concentrations, since sharp cracks also intersected the free surface adjacent to cracks which were associated with pits. Presumably these cracks formed late in specimen life since they are not extensively pitted (Fig. 14-b and c). Additionally, the blunted nature of the cracks on the specimen surface suggests that the cracks were broadened by a corrosion reaction.

### Discussion

The results of this study show that hydrogen, either produced by cathodic charging or by corrosion reactions, lowers the resistance of 7075 Al to cyclic deformation. Additionally, it has been shown that, under conditions of free corrosion, the anionic species is important to the fatigue process,  $\text{Cl}^-$  being considerably more damaging than  $\text{SO}_4$  of the same ionic concentration. The application of cathodic currents in either  $\text{Cl}^-$  or  $\text{SO}_4^{=}$  solution on the other hand results in a similar reduction in fatigue resistance, particularly at applied stresses in the vicinity of the  $10^7$ -cycle fatigue limit. It has also been shown that a cathodic poison (As) reduces fatigue resistance in  $\text{Cl}^-$  solutions and that acid solutions are more damaging than basic solution of the same aggressiveness (as measured by overall corrosion rate).

Correlation of the fatigue resistance results with electron fractography and optical metallography indicates that hydrogen induces a form of cyclic cleavage in high strength aluminum alloys which is characterized by crack initiation on a plane normal to the tensile axis. This zone, which is essentially featureless, transforms to a zone of quasi-cleavage with a considerably more brittle appearance than is observed when fatigue crack propagation occurs in laboratory air. This region transforms to the feathery river line appearance similar to that of cracks propagated in air as the crack grows.



The featureless and quasi-cleavage zones are observed under all conditions of environmental fatigue, including tests performed at pH 2, pH 10.2 and under conditions of a small applied anodic potential. It should be pointed out, however, that even at pH 10.2, the corrosion potential of the alloy is more active than the reversible hydrogen electrode potential ( $\phi_{\text{corr}} = -.96\text{v}_{\text{SHE}}$ ,  $\phi_{\text{H}_2} = -0.6\text{v}$  vs. SHE. Thus, thermodynamically, atomic hydrogen can be generated even at this high pH. Additionally, after the crack has initiated, it is likely that the pH within the growing crack is actually more acid than the bulk solution, further providing  $\text{H}_2$  to the crack tip.

It is interesting to note that, in sulfate, externally generated hydrogen (in contrast to  $\text{H}_2$  generated from an intrinsic corrosion reaction) results in almost the same fatigue resistance as is observed in  $\text{Cl}^-$  solutions. At relatively high stress levels, cathodic charging in the sulfate solution shows a slightly superior fatigue resistance while at stresses near the  $10^7$  cycle fatigue limit fatigue resistance is virtually identical. Since  $\text{Cl}^-$  is known to attack the protective film formed on Al alloys in neutral environments, it is likely that, at high stresses the film damage allows better access of the hydrogen to the alloy and accordingly lowers the crack initiation time. At low stresses, crack initiation time and initial crack propagation rates are slow so that there is ample time for the hydrogen to diffuse into the

surface of the alloy and to the tip of a small growing crack without necessitating film damage by  $\text{Cl}^-$ . This hypothesis is further supported by the observation that cathodic charging at high stresses in the  $\text{SO}_4^{=}$  solution does not affect fatigue resistance (relative to free corrosion in  $\text{SO}_4^{=}$ ), probably because the crack propagation rate is sufficiently large to prevent an equivalent  $\text{H}_2$  concentration in the vicinity of a growing crack tip. In chloride solutions, on the other hand, there is a slight improvement of fatigue resistance at high stresses and a reduction at lower stresses. Since it is believed that, while free corrosion is occurring, hydrogen is evolved in the vicinity of preferentially corroded pits and in a growing crack<sup>11</sup>, the increased resistance at high stresses may be due to a reduction in  $\text{H}_2$  in a growing crack due to some anodic polarization of the crevice created by the growing crack. At low stresses, crack initiation and early propagation are the limiting rate factors and the excess hydrogen produced by cathodic polarization serves to shorten the time required for these processes.

It is interesting, although probably not surprising, to note that the T73 heat-treatment does not improve corrosion fatigue resistance in  $\text{Cl}^-$  resistance. Since the T73 heat-treatment was developed to provide intergranular SCC resistance by altering grain boundary area structures, while the corrosion fatigue process is transgranular, no beneficial effect should be expected.



The fractography results support the view that a form of hydrogen embrittlement is responsible for the reduction in fatigue resistance observed in this study since the featureless and cleavage like zones could result from local embrittlement of the alloy.

It has been suggested that aluminum alloys should not be expected to be susceptible to hydrogen embrittlement because of the low diffusivity of hydrogen in aluminum. However, the effect of hydrogen on fatigue behavior of aluminum alloys does not depend strongly on bulk diffusivity of hydrogen but only requires that hydrogen be present in the vicinity of the growing crack, i.e., in the plastic zone. In this region fresh metal is constantly being exposed to the environment. Additionally dislocations associated with the plastic zone may act as high diffusivity paths for hydrogen, thus locally increasing the hydrogen concentration<sup>12,13</sup>. At rapid crack propagation rates the crack would be expected to grow more rapidly than a "critical" (as yet undefined) concentration of hydrogen can be developed in the vicinity of the crack tip, and the fracture surface appearance will resemble that observed in air. An additional factor is that it has been shown that cathodic charging of a surface containing a crack or crevice can result in anodic polarization of the metal or alloy in the defect<sup>11</sup>. Thus less hydrogen would be available for embrittlement as the crack becomes longer. This suggestion may explain the constant life for decreasing potentials below some critical value, since the production of



hydrogen at the surface would not greatly affect conditions in the crack at long distances from the free surface.

The specific mechanisms of embrittlement is still not completely understood, although it may be associated with a reduction in plasticity at the crack tip by a Cottrell atmosphere phenomenon. For example, recent tensile tests conducted on a high purity analogue of the 7075 alloy showed serrated yielding of cathodically polarized specimens, suggesting a dislocation-solute<sup>12</sup> interaction. This observation, of a hydrogen dislocation interaction, also supports the hypothesis that dislocations in the plastic zone ahead of a growing crack may act as high diffusivity paths for hydrogen.

#### Summary and Conclusions

1. Neutral sulphate solutions under free corrosion conditions affect corrosion fatigue of 7075 Al alloys to approximately the same degree as distilled  $H_2O$ . Both solutions lower fatigue resistance when compared to laboratory air. Neutral chloride solutions are considerably more aggressive under free corrosion conditions.
2. Corrosion fatigue behavior of 7075 Al is not directly coupled to general corrosion rate, acid solutions reducing fatigue resistance more than basic solutions of the same corrosivity.

3. Cathodic charging of 7075 Al results in a dramatic reduction in fatigue resistance in either sulfate or chloride solutions. This observation, coupled with fractographic evidence of cyclic cleavage, indicates that a form of hydrogen embrittlement is responsible for corrosion fatigue of this alloy.

4. There is no inherent advantage to the T73 temper for resistance to corrosion fatigue in smooth surface specimens.

The results of this investigation suggest that dissolved hydrogen need only interact with the surface at nascent cracks and in the plastic zone of growing cracks, which accounts for the embrittlement effect in cyclic deformation experiments which is not observed in smooth surfaced, static deformation experiments.

#### Acknowledgments

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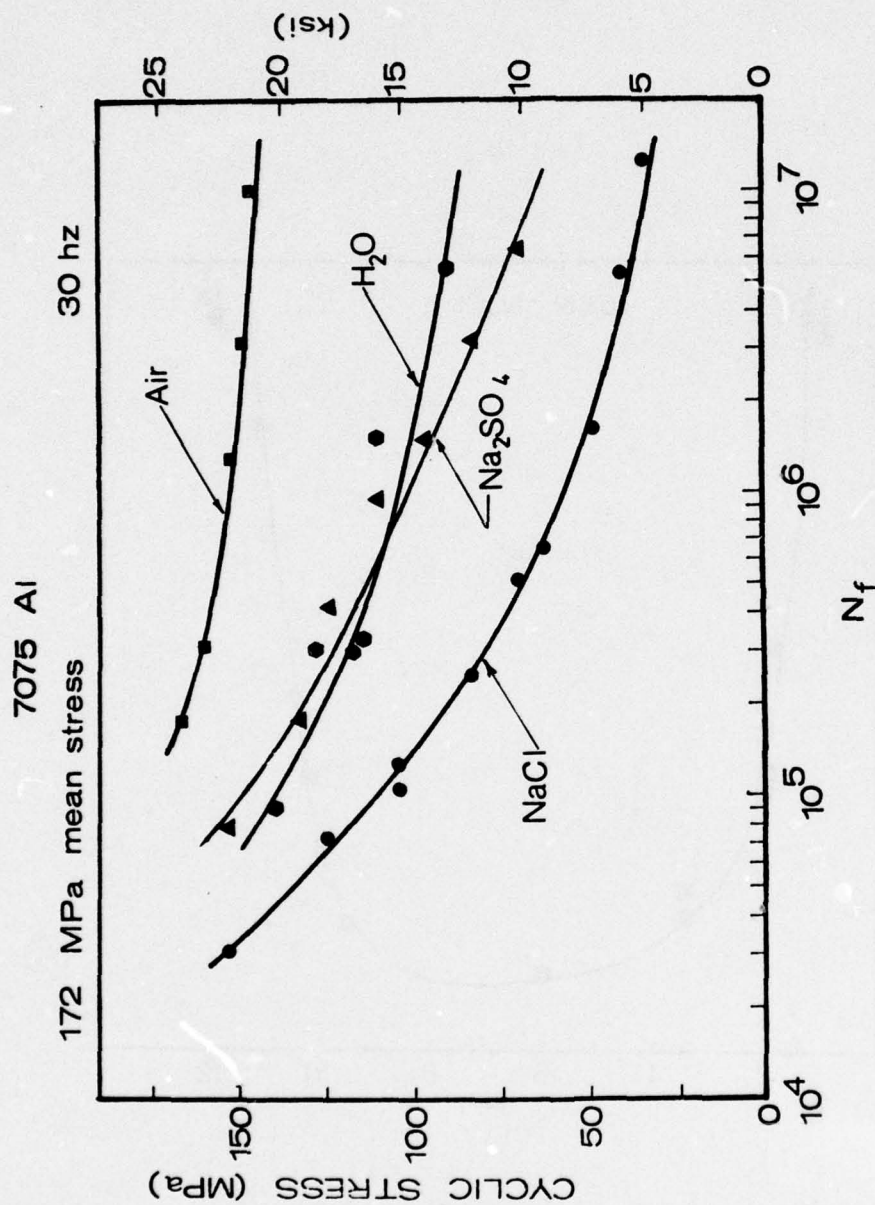


Fig. 1 Fatigue behavior of 7075-T6 Al alloy in air, distilled H<sub>2</sub>O and aqueous solutions of 0.5N Na<sub>2</sub>SO<sub>4</sub> and 0.5N NaCl.



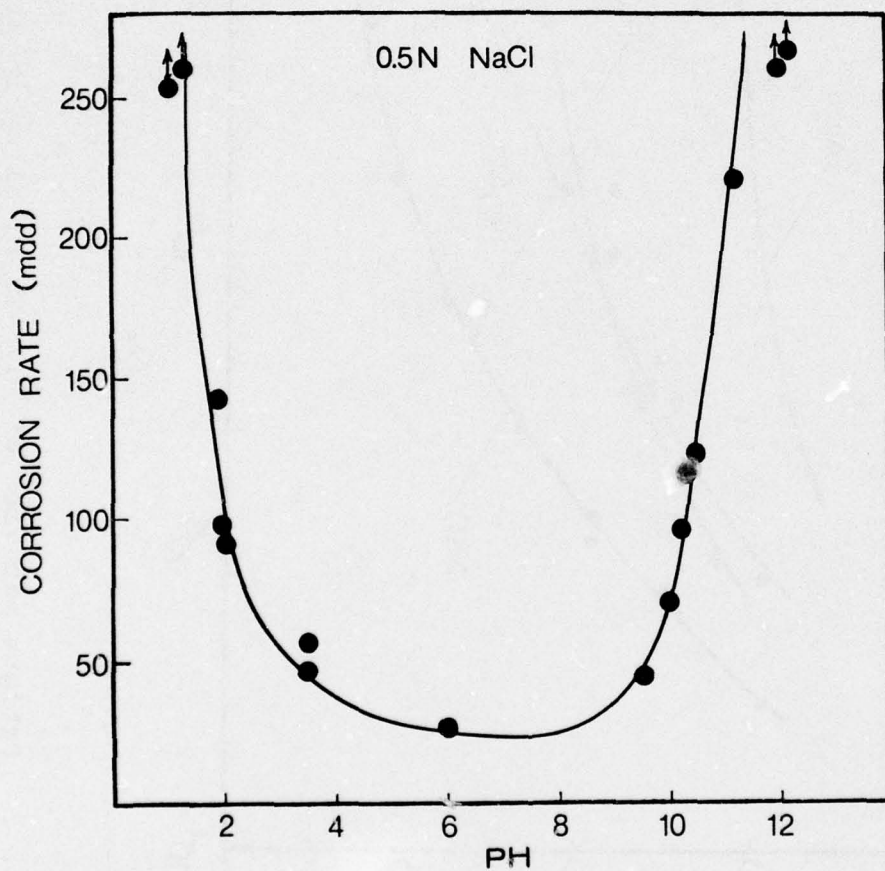


Fig. 2 The effect of pH on general corrosion of unstressed 7075-T6 alloy in aqueous 0.5N NaCl solution (1mdd = 2cm/yr overall penetration).

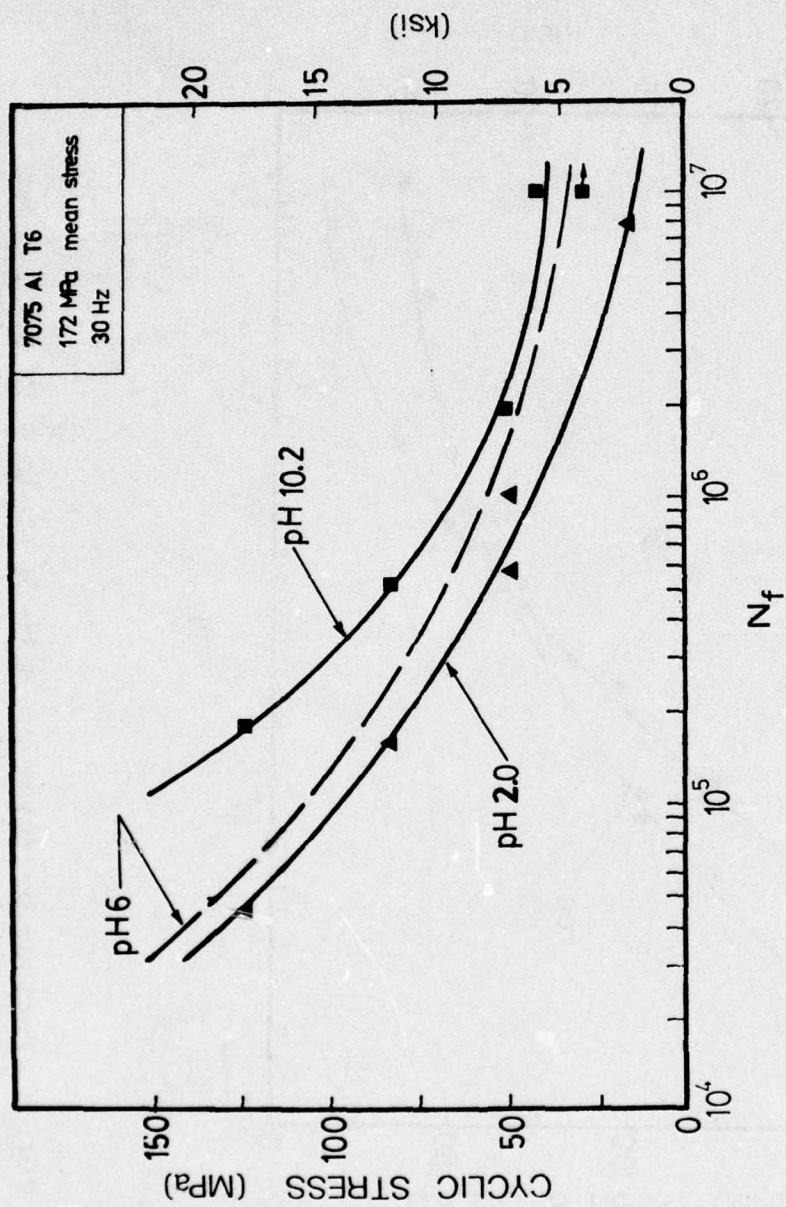


Fig. 3 Fatigue behavior of 7075-T6 Al in 0.5N NaCl solutions of pH 2 and pH 10.2; pH 6 data is repeated from Fig. 1.



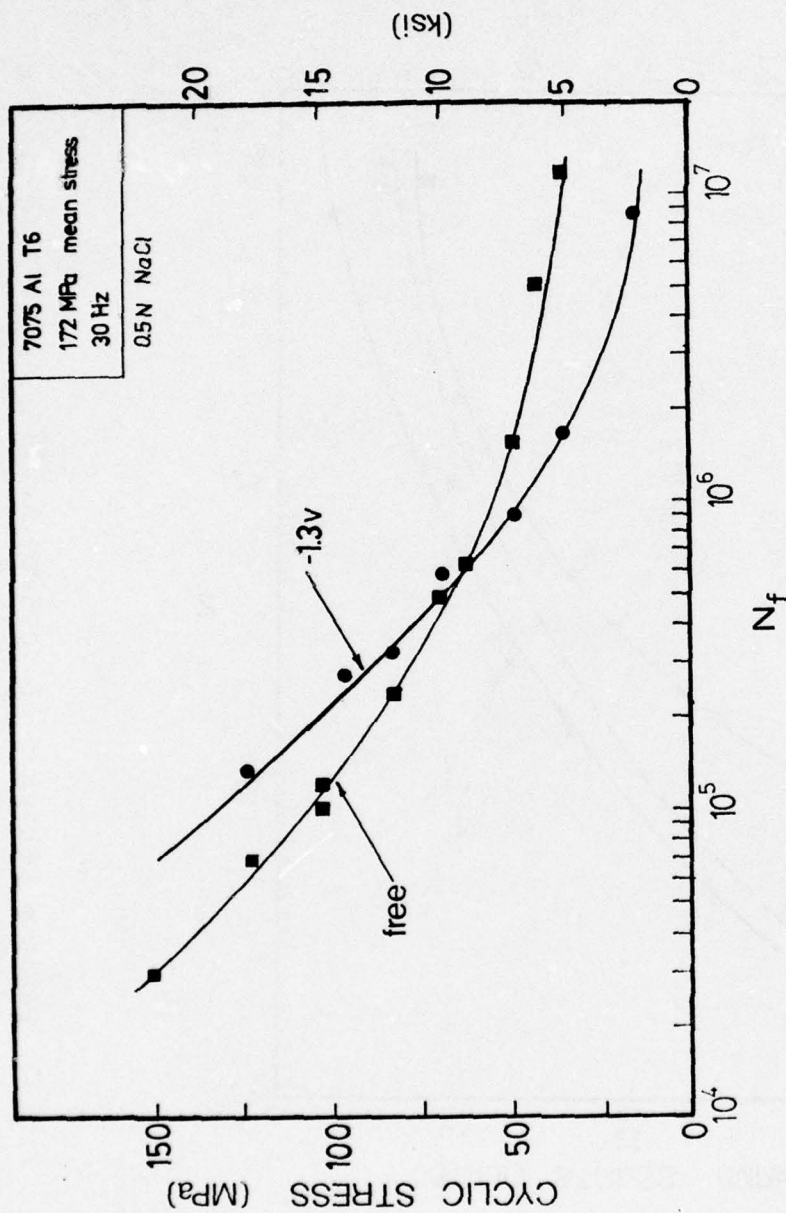


Fig. 4 Fatigue behavior of 7075-T6 Al in 0.5N NaCl under conditions of cathodic charging at  $\phi_c = -1300$  mv vs.SCE.

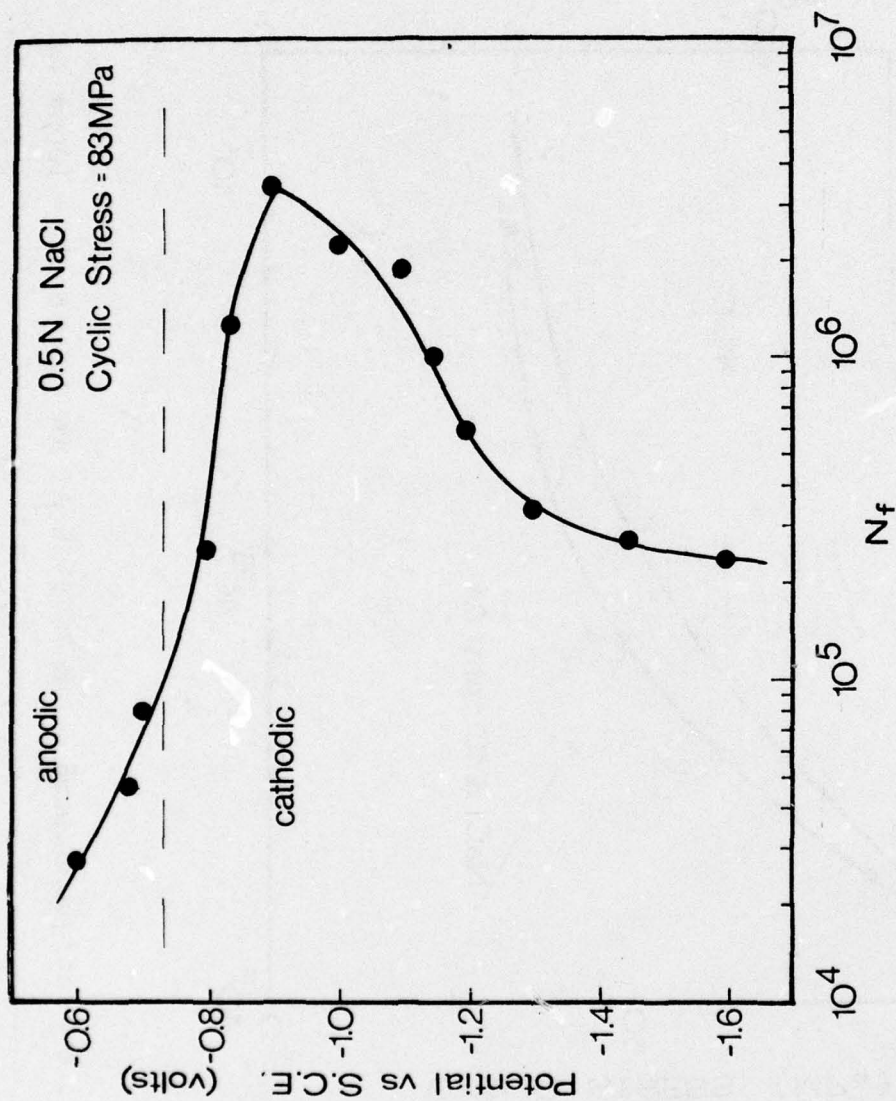


Fig. 5 Fatigue life of 7075-T6 Al in 0.5N NaCl under conditions of cathodic charging,  $\sigma_{\max} = 255$  MPa.



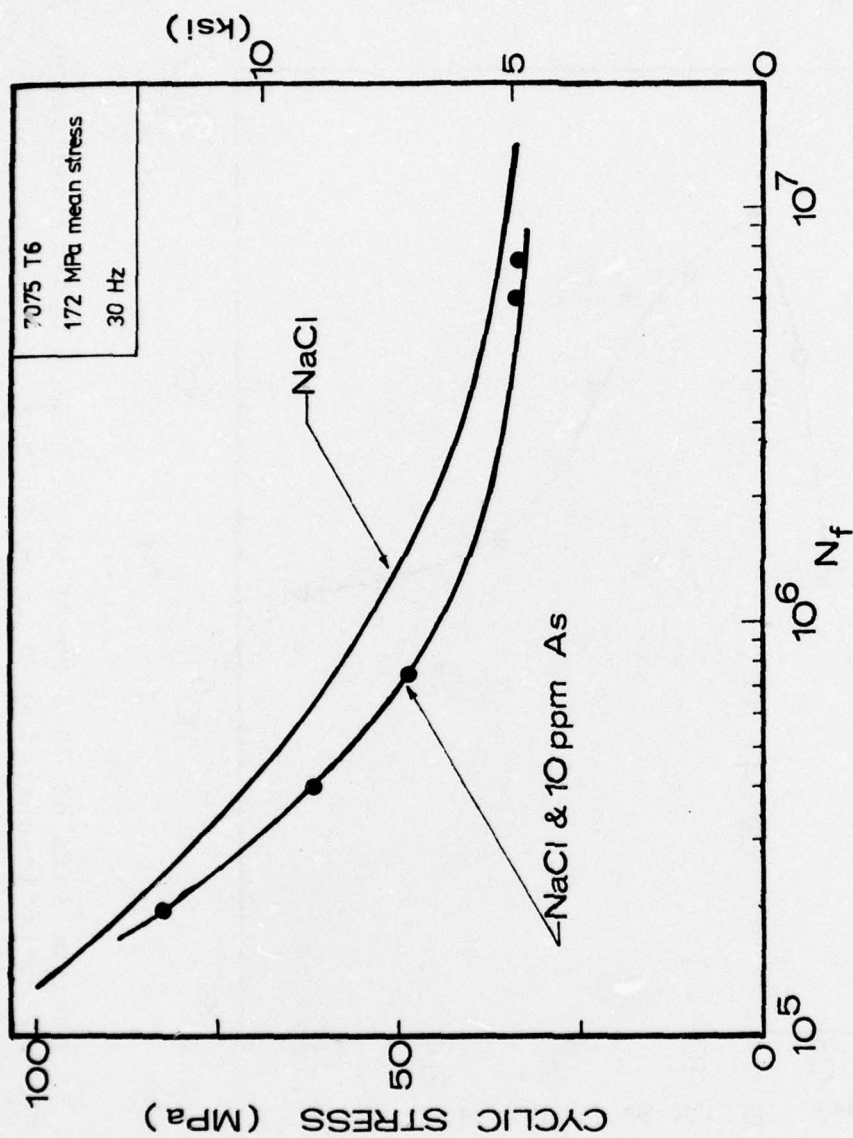


Fig. 6 Fatigue behavior of 7075-T6 Al in 0.5N NaCl + 10ppm As.

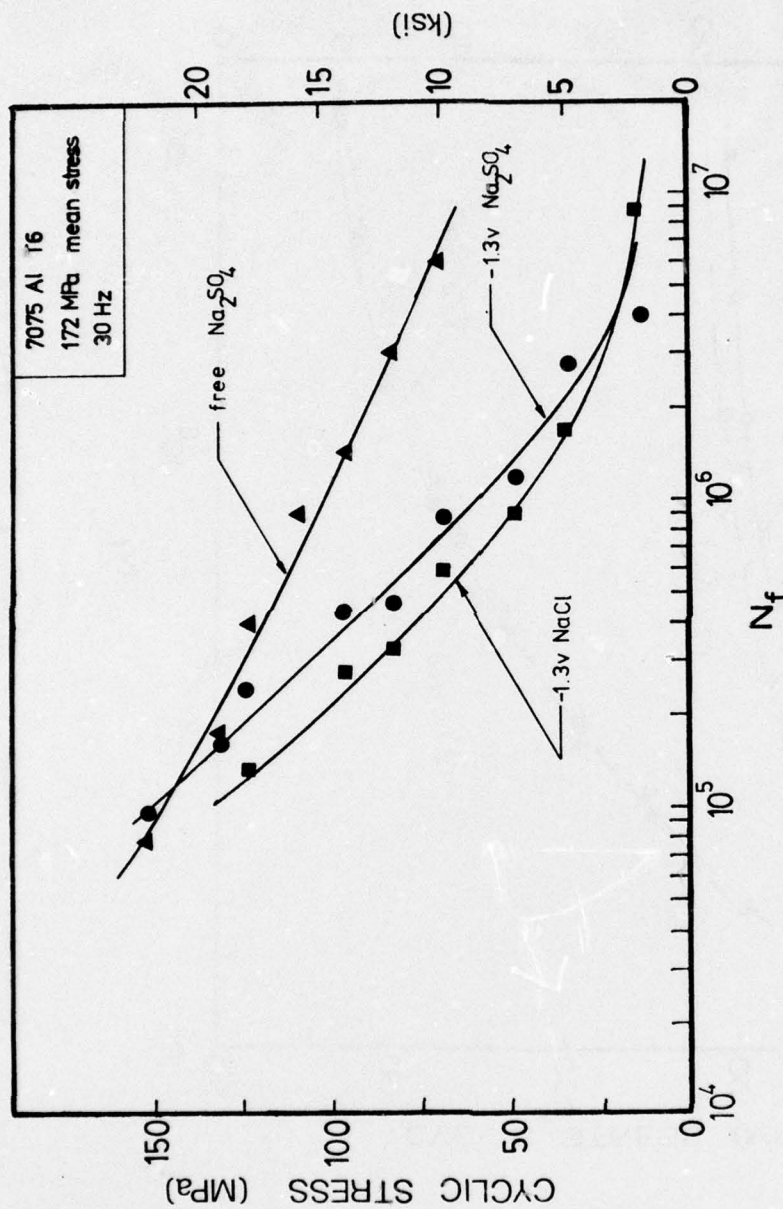


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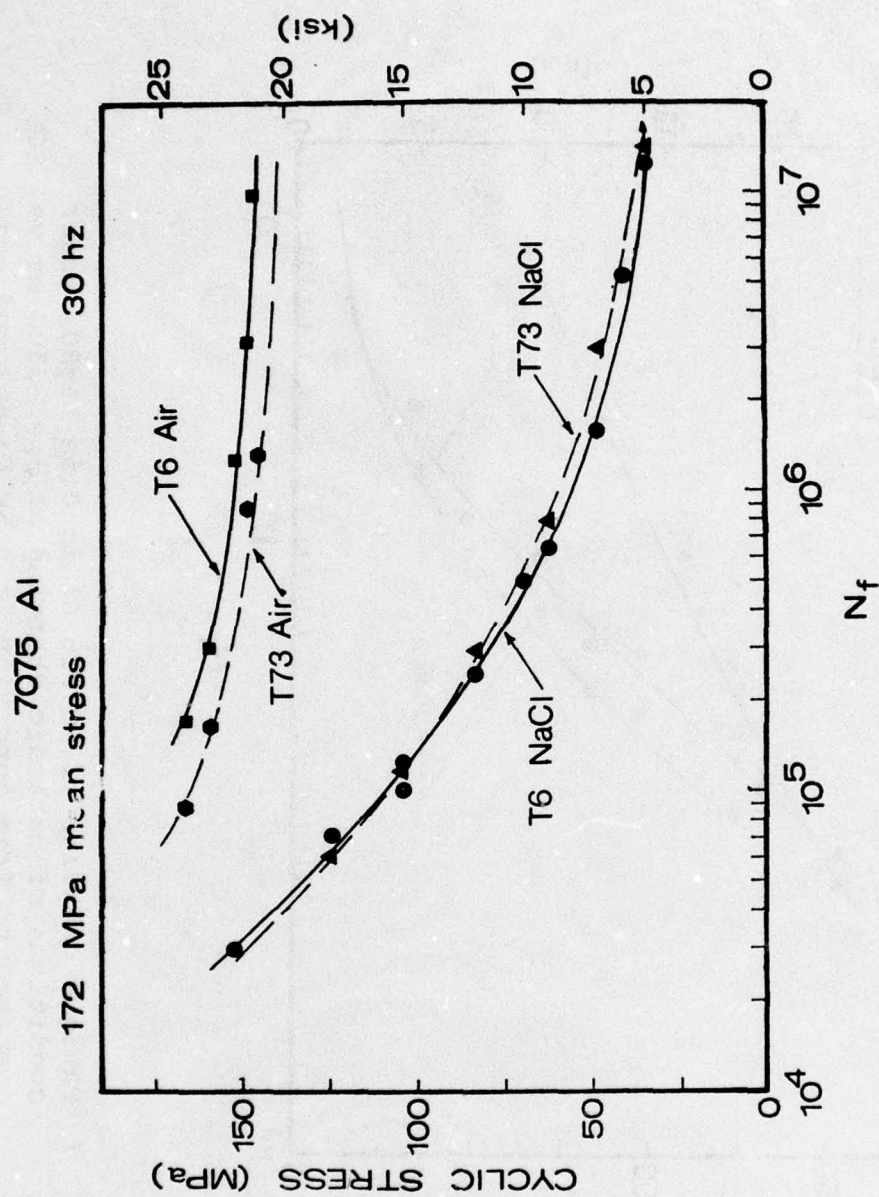
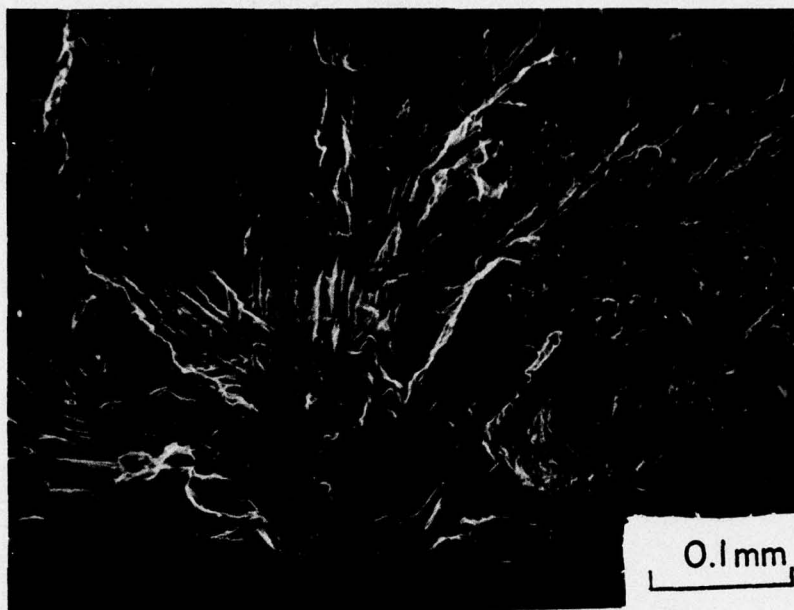
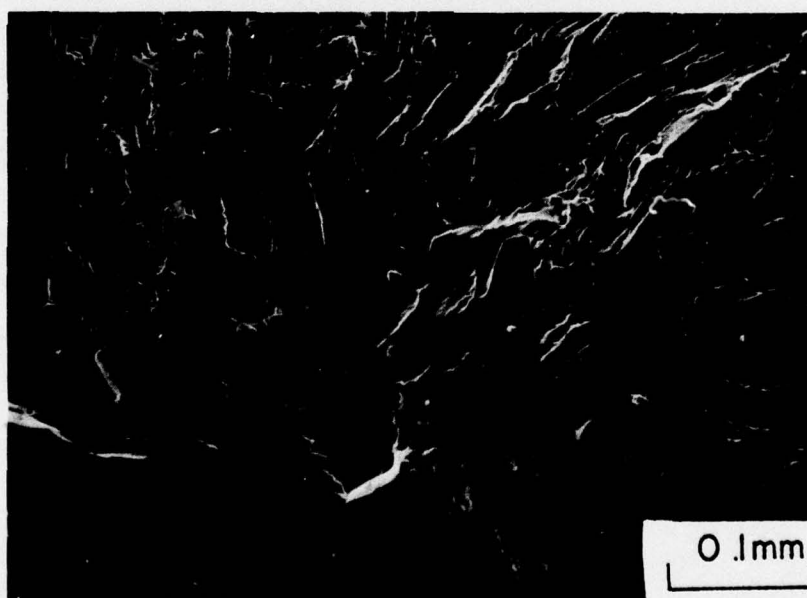


Fig. 8 Fatigue behavior of 7075-T73 Al in air and in 0.5N NaCl compared to behavior of 7075-T6 Al.



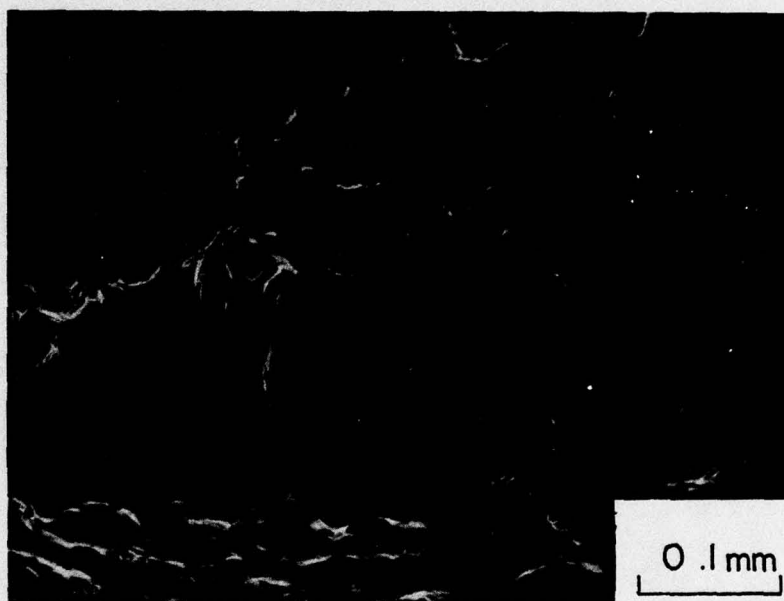
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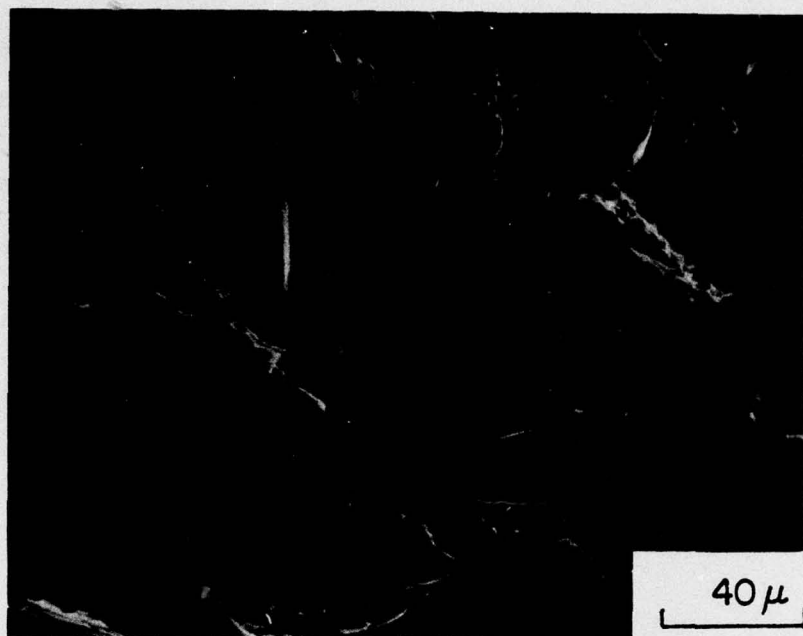
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Fig. 9 Fracture surfaces of 7075-T6 Al in (a) air, (b) freely corroding 0.5N NaCl solution and (c) 0.5N NaCl solution under conditions of cathodic charging at  $\phi_c = -1300$  mv vs. SCE,  $N_f = 10^6$ .

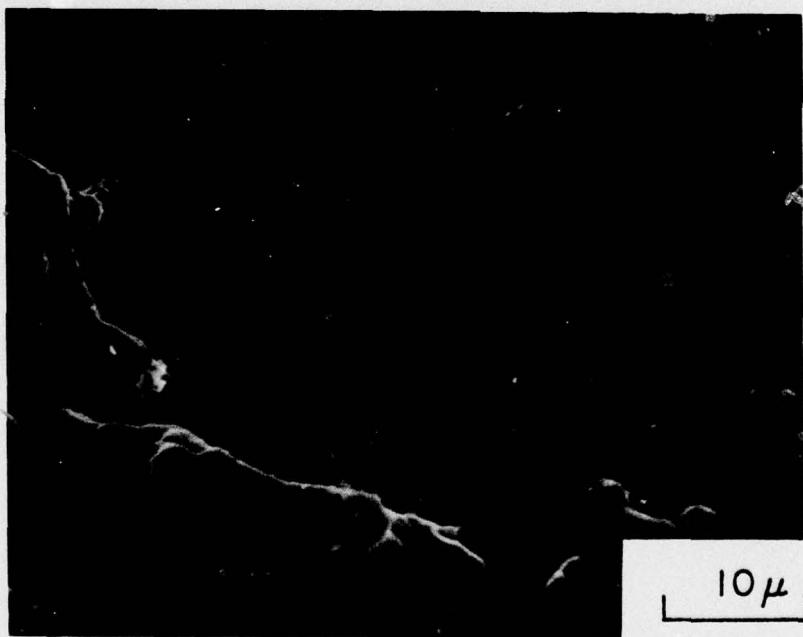




(c)



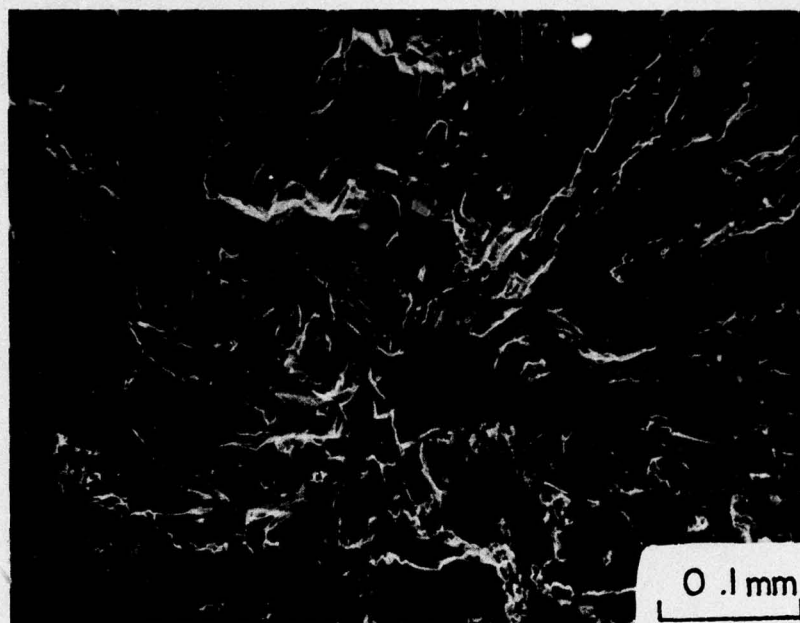
(a)



(b)

Fig. 10 Fracture surface of 7075-T6 Al in 0.5N  $\text{Na}_2\text{SO}_4$  solution  $N_f = 5 \times 10^5$ .





(a)



(b)

Fig. 11 Fracture surfaces of 7075-T6 Al in 0.5N NaCl of (a) pH 2 and (b) pH 10.2,  $N_f = 10^6$ .

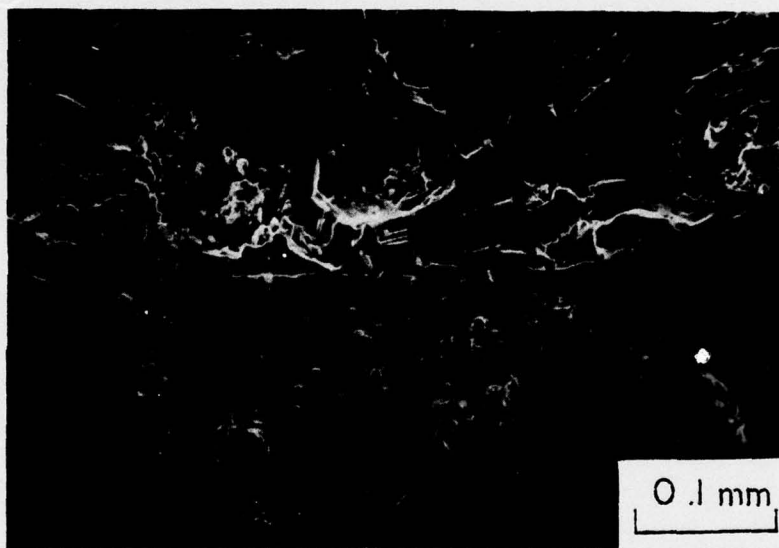
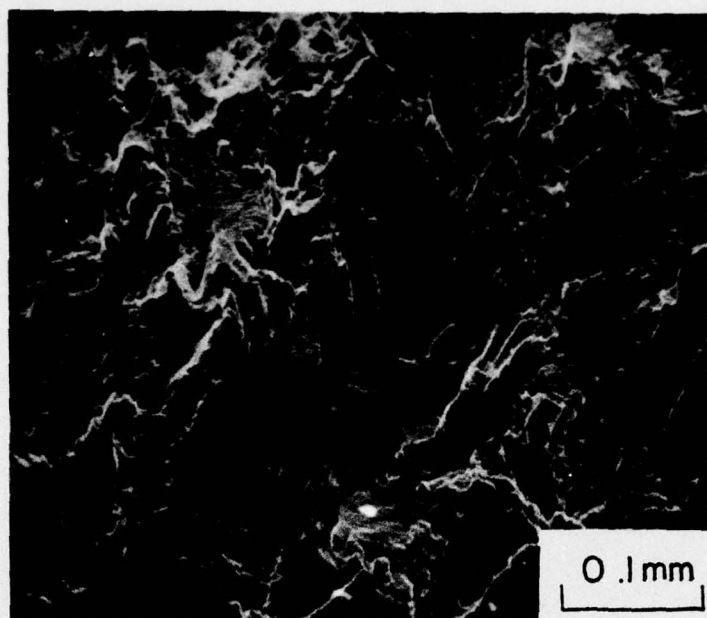
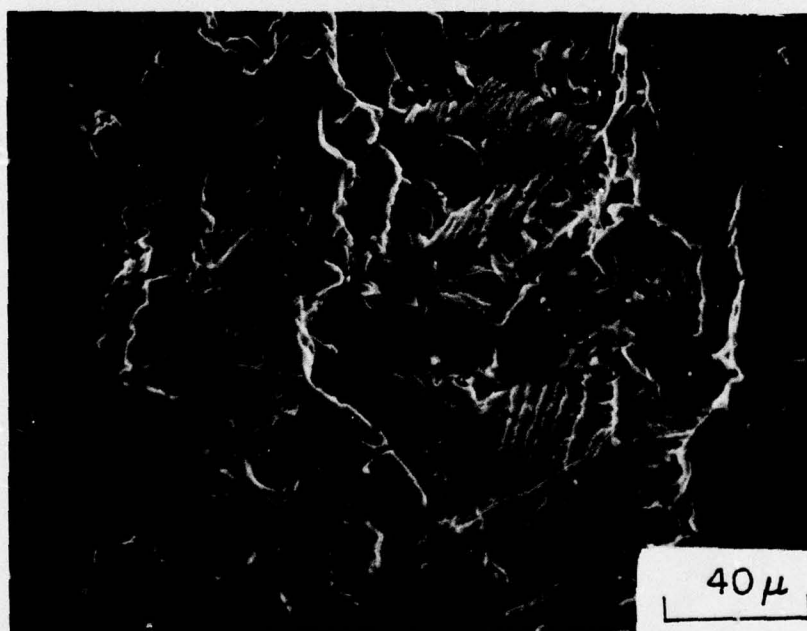


Fig. 12 Surface pitting of 7075-T6 Al in 0.5N NaCl solution of pH2.



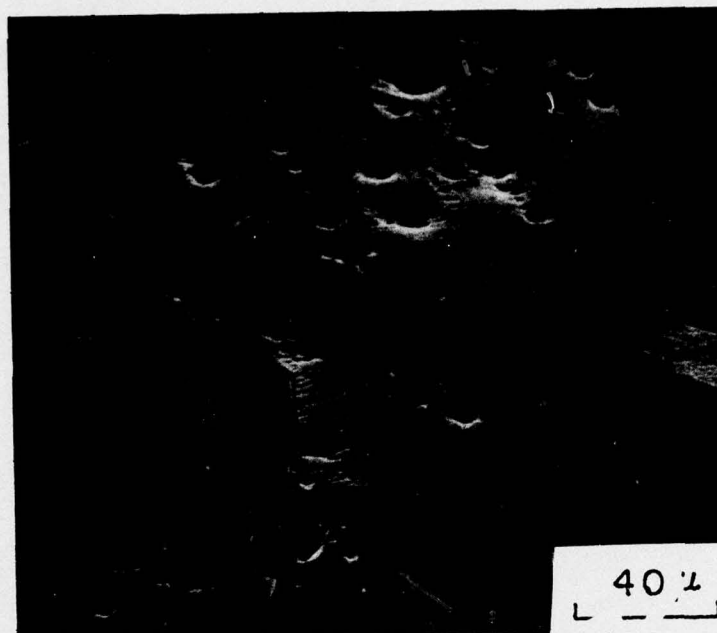


(a)

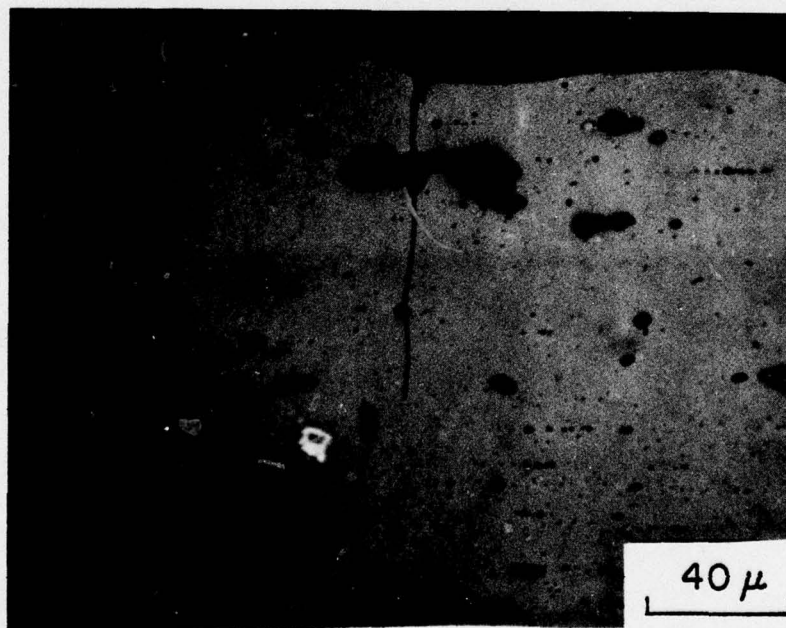


(b)

**Fig. 13** Fracture surface of 7075-T7 Al in 0.5N NaCl under conditions of cathodic charging (a)  $\phi_c = -1300$  mv vs. SCE showing Stage II cracking and (b)  $\phi_c = -900$  mv vs. SCE showing intergranular corrosion.

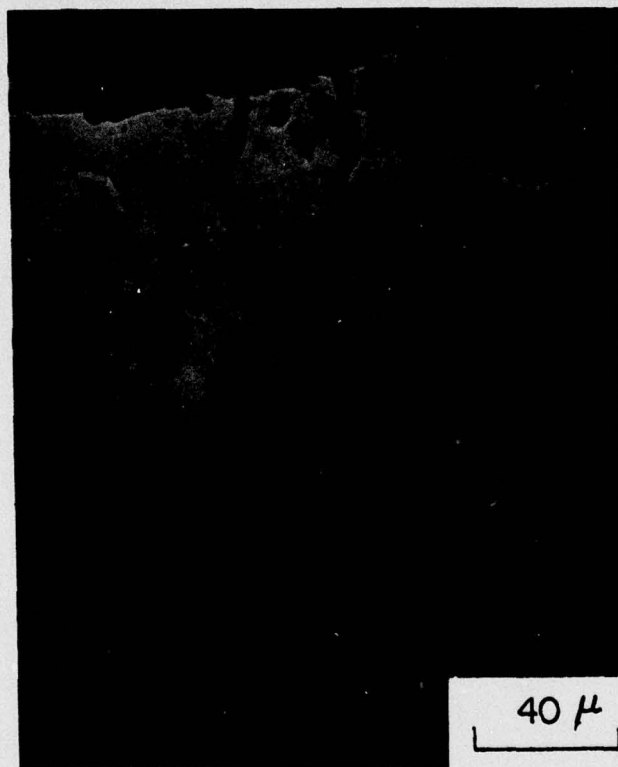


(a)



(b)

Fig. 14 (a) Secondary cracking and pitting of 7075-T6 Al alloy in 0.5N NaCl under conditions of cathodic charging at  $\phi_c = -1300$  mv vs. SCE, (b and c) optical micrographs of a cross-section of 7075-T6 alloy showing secondary cracking and pits associated with secondary cracks under cathodic charging conditions.



(c)



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13. ABSTRACT High cycle fatigue experiments have been performed on a 7075 Al alloy, principally in the T6 temper in dry air, distilled water 0.5N NaCl and 0.5N Na <sub>2</sub> SO <sub>4</sub> solutions as functions of pH, cathodic charging and catalyst poisoning of the hydrogen evolution reaction. All aqueous solutions appreciably lowered fatigue resistance with Cl <sup>-</sup> ion producing the greatest reduction in resistance and SO <sub>4</sub> <sup>=</sup> ion behaving essentially in the same manner as distilled water. Under cathodic charging conditions fatigue resistance is significantly reduced and both Cl <sup>-</sup> and SO <sub>4</sub> <sup>=</sup> solutions produce similar fatigue lives. Acid solutions reduce fatigue resistance to a greater degree than do basic solutions of equivalent corrosivity and a catalyst poison (As) added to Cl <sup>-</sup> solutions reduces fatigue resistance relative to neutral Cl <sup>-</sup> solutions. Fractography of specimens fatigued in aqueous environments shows that a significant amount of cleavage and quasi-cleavage occurs, the extent of these features being apparently a function of available hydrogen to the alloy free surface and to the tips of growing cracks. On the basis of these observations, it is suggested that corrosion fatigue of 7075 alloy is essentially a hydrogen embrittlement phenomenon where the low diffusivity of hydrogen is counterbalanced by the fact hydrogen need only be present in the alloy free surface for crack initiation and in the plastic zone of growing cracks for propagation.			

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